Effect of light curing unit and antiseptic mouthwashes on the physical properties of gingiva-colored composite resin

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ABSTRACT | *Objective*: This study evaluated surface roughness (Ra) and color difference (ΔE_{00}) of a gingiva-colored composite resin (NT Premium Gengiva, Coltene), light-activated with three light curing units (LCU) and immersed in different mouthwash solutions. *Methods*: 90 composite specimens (n=10) were divided according to the LCU: L1-Valo, L2-Bluephase and L3-Optilight Max; and solution: S1-alcohol-based antiseptic mouthwash (Listerine Cool Mint), S2-alcohol-free antiseptic mouthwash (Listerine Cool Mint Zero Alcohol) and S3-water (control). Immersions were performed for 30 s, 2x/day, for 21 days. Ra and coordinates of luminosity (L*) and color (a* and b*) were measured using a rugosimeter (SJ-201 P/M) and a spectrophotometer (SP62S), respectively, at: T0-start, T1-7 days, T2-14 days and T3-21 days. *Results*: L1 (1.3±0.9) presented higher Ra than L3 (0.7±0.6) and L2 (1.0±0.8); and Ra for S2 (0.9±0.1) was greater than S3 (-0.1±0.1), and S1 (0.1±0.1). Moreover, L1 (23.3±0.3) presented lower ΔE_{00} than L2 (24.7±0.3) and L3 (25.1±0.3). *Conclusion*: Ra of the gingiva-colored composite resin tested increased when L1 and S2 were used, although L1-cured specimens also showed the lowest ΔE_{00}.

DESCRIPTORS | Composite Resins; Light Curing; Mouthwashes.

RESUMO | **Efeito da unidade fotopolimerizadora e enxaguantes bucais antissépticos nas propriedades físicas de um compósito de resina cor de gengiva** • *Objetivo*: Este estudo avaliou a rugosidade superficial (Ra) e a diferença de cor (ΔE_{00}) de um compósito de resina composta cor de gengiva (NT Premium Gengiva, Coltene), fotopolimerizável com três unidades fotopolimerizáveis (UFP) e imersa em diferentes soluções de enxaguantes. *Métodos:* Ao todo, 90 espécimes de compósitos (n=10) foram divididos de acordo com o UFP: L1-Valo, L2-Bluephase e L3-Optilight Max; e solução: S1-enxaguante bucal antisséptico à base de álcool (Listerine Cool Mint), S2-enxaguante bucal antisséptico sem álcool (Listerine Cool Mint Zero Alcohol) e S3-água (controle). As imersões foram realizadas por 30 s, 2×/dia, por 21 dias. Ra e coordenadas de luminosidade (L*) e cor (a* e b*) foram medidas usando um rugosímetro (SJ-201 P/M) e um espectrofotômetro (SP62S), respectivamente, em: T0-início, T1-7 dias, T2-14 dias e T3-21 dias. *Resultados:* L1 (1,3±0,9) apresentou Ra maior que L3 (0,7±0,6) e L2 (1,0±0,8); e Ra para S2 (0,9±0,1) foi maior do que S3 (-0,1±0,1) e S1 (0,1±0,1). Além disso, L1 (23,3±0,3) apresentou ΔE_{00} menor que L2 (24,7±0,3) e L3 (25,1±0,3). *Conclusão*: O Ra do compósito de resina cor de gengiva testado aumentou quando L1 e S2 foram usados, embora os espécimes curados com L1 também tenham apresentado o menor ΔE_{00}.

DESCRITORES | Compósito de resina; Fotopolimerizador; Enxaguantes bucais.

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INTRODUCTION

Gingival recession is defined as the exposure of root surface due to migration of the apical gingival margin towards the amelo-cemental junction (ACJ).[1] It is a concern of patients and professionals and in some cases may be associated with cervical lesions caused by cavities, abrasion or erosion.[2,3]

Treatment options include using gingiva-colored composite resin (GR) to simulate gum tissue.[4,5] Pioneered by Zalkind and Hochman,[6] this type of restoration material is used in treatments that correct cervical defects, radicular erosions, abfraction lesions, Class V lesions (cervical caries) and in cases of periodontal recession to reduce hypersensitive or darkened tooth gumlines.[4,6-8]

The correct polymerization can increase the clinical longevity expectancy of composite resins; this is achieved by using a light curing unit (LCU) to control the intensity, wavelength and duration of light exposure. Inadequate polymerization can result in marginal microinfiltration due to bonding defects, with a corresponding decrease in performance regarding the physical and mechanical properties of the composite resin.[9,10]

Antiseptic mouthwash use has grown in recent years, contributing to control and reduce plaque and gingivitis.[11] On the other hand, mouthwashes can negatively impact the durability of restoration materials. This is the case for both alcohol and alcohol-free versions since alcohol is only one of several factors that can result in alterations.[11] The effect that these antiseptics can have on wear and hardness will depend on the type of material and chemical composition, type and filler content.[11]

Chemical degradation of restored surfaces is caused by interactions between several chemical compounds. The different negative effects of antiseptic mouthwash products are expected due to the presence of different solutions (e.g., water and alcohol) alongside factors such as a low pH. Few antiseptic mouthwash products present pH lower than 5.5. Consequently, it is important to study their effects on restored surfaces.[12-14]

Studies[15,16] have demonstrated positive results regarding aesthetics of materials that simulate gum tissue along the cervical margins such as GR, receiving positive evaluation from patients. Conversely, there is a lack of studies that focus on an in-depth exploration of the properties of these materials. This study seeks to fill this gap and present an in vitro investigation that analyzes surface roughness (Ra) and color difference (ΔE_{00}) of a GR light-activated by different LCU and immersed in different mouthwash solutions for different time durations.

MATERIALS AND METHODS

Materials

Materials and methods used throughout this study and their specifications are described in Tables 1 and 2.

Table 1 | Restoration Materials and Solutions

Material	Composition	Manufacturer
NT Premium Gum	BisGMA, Bis-EMA, TEGDMA, UDMA, BHT, photoinitiator, charges and pigments	Coltene
Antiseptic mouthwash Listerine Cool Mint	Water, Sorbitol, Alcohol (21,6%), Poloxamer 407, Benzoic Acid, Eucaliptol, Sodium Saccharin, Scent agent (D-limonene), Timol, Methyl Salicylate, Sodium Benzoate, Menthol, CI 42053	Johnson & Johnson
Antiseptic mouthwash Listerine Cool Mint Zero Alcohol	Water, Sorbitol, Poloxamer 407, Benzoic Acid, Eucaliptol, Propylene glycol, Lauryl Sodium Sulphate	Johnson & Johnson
Natural mineral water Minalice	Strontium, Calcium, Magnesium, Potassio, Sodium, Vanadium, Sulfate, Carbonate, Biocarbonate, Fluoride, Chloride	Minalice Mining Company LTDA

Table 2 | Light Curing

	Light Curing	Power output	Wavelength	Manufacturer
VALO	LED	1400 mW/cm2	395 – 480 nm	Ultradent
Bluephase	LED	1200 mW/cm2	385 – 515 nm	Ivoclar Vivavent
Optilight Max	LED	≥ 1200 mW/cm2	420 – 480 nm	Gnatus

Experimental design

The study variables in this experiment are: Ra and ΔE_{00}. The variation factors are: (1) Mouthwash solutions [S1 – alcohol-based mouthwash (Listerine Cool Mint – Johnson & Johnson, São Paulo, Brazil); S2 – alcohol-free mouthwash (Listerine Cool Mint Zero Alcohol – Johnson & Johnson, São Paulo, Brazil) and S3 – natural mineral water (Minalice Mining Company LTDA – São Paulo, Brazil)]; (2) LCU: [3 levels: L1 –Valo (Ultradent Products Inc – South Jordan, USA); L2 – BluePhase (Ivoclar Vivadent AG – Schaan, Liechtenstein) and L3 – Optilight Max (Gnatus – Ribeirão Preto, São Paulo, Brazil)]; and mouthwash solution application time [4 levels: 48 h (T0), 7 (T1), 14 (T2) and 21 (T3) days after specimens were obtained].

Sample preparation

In total, 90 specimens of a GR (NT Premium Gum, Coltene, Rio de Janeiro, Brazil) were obtained (n=10) according to manufacturer's instructions using a teflon matrix of 6 mm diameter and 2 mm depth on a glass plate and a polyester matrix.

The material was inserted in the matrix in a single increment with the aid of a resin spatula (Duflex, São Paulo, Brazil). The filled cavity was covered by a polyester matrix and a glass plate. Following, a 1 kg weight was put on top to guarantee the complete filling of the matrix and cause any excess material to overflow. Next, light activation was applied on the tested specimens using the previously mentioned devices (Table 2), in accordance with instructions from the manufacturer.

After the photopolymerization, samples were taken out of the matrix and kept in artificial saliva for 24 hours inside an oven at 37±1 °C. Following, each specimen was polished with Sof Lex (3M, São Paulo, Brazil) discs in decreasing grit sequence, wetting the specimen surface between applications to avoid overheating and the consequent alteration of the surface. One of the surfaces of each specimen was marked to serve as a positioning guide to be used with the rugosimeter and the spectrophotometer.

Sample immersion

Samples were stored in artificial saliva composed of methylparaben (2.0), sodium carboxymethylcellulose (10.0 g), KCl (0.625 g), MgCl 2.6H2O (0.059 g) CaCl2-2H2O (0.166 g), K2HPO4 (0.804 g), KH2PO4 (0.326 g) in 1000 mL of distilled water inside an oven at 37±1 °C during the entire experiment. The artificial saliva was changed daily and only taken out to be exposed to the solutions and to take measurements at 48 hours, 7, 14 and 21 days.

Each specimen was immersed individually in a container with 1 ml of each test solution (Table 1) for 30 seconds, simulating daily rinsing with vibration, following instructions by the manufacturer (Vibramaxx Gold Line platform shaker – Essence Dental VH, Araraquara, São Paulo, Brazil) twice per day, for 21 days.

Ra measurement

Ra measurements were obtained using a rugosimeter (SJ-201 P/M, Mitutoyo, Tokyo, Japan). Each specimen was dried with paper towel and positioned on the

device using tweezers (Colgran, São Paulo, Brazil); three readings were taken to register the mean value. Ra was performed at initial (48 hours after test specimens were created), 7 days, 14 days and 21 days.

ΔE_{00} measurement

ΔE_{00} was measured using an SP62S spectrophotometer and the QA-Master I software (X-Rite Incorporated, Neu-Isenburg, Germany). Each specimen was carefully manipulated using tweezers (Millennium, Golgran, São Paulo, Brazil), dried with a paper towel and kept in a device prepared to hold the samples and take readings with white and opaque background. The color (ΔE_{00}), lightness ($\Delta L'$), chroma ($\Delta C'$), and hue differences ($\Delta H'$) resulting of the specimens light activated with different LCU and immersed in different mouthwash solutions were calculated by the CIEDE2000 formula.[17,18]

$$\Delta E_{00} = \left[\left(\frac{\Delta L'}{K_L S_L}\right)^2 + \left(\frac{\Delta C'}{K_C S_C}\right)^2 + \left(\frac{\Delta H'}{K_H S_{LH}}\right)^2 + R_T\left(\frac{\Delta C'}{K_C S_C}\right)\left(\frac{\Delta H'}{K_H S_H}\right)\right]^{\frac{1}{2}}$$

Where R_T is a rotation function; S_L, S_C, and S_H are weighting functions; and the parametric factors K_L, K_C and K_H are terms to be adjusted, which in the present study were set to 1. This study used a general average of the L*a*b* coordinates of the unrepaired group (BCR) (L* = 72.6; a* = 4.7; b* = 7.6) as the reference mean for experimental comparisons.[17,18] The color variation measurements were performed at initial (48 hours after test specimens were created), 7 days, 14 days and 21 days.

Statistical analysis

Ra and ΔE_{00} data were analyzed using the Shapiro-Wilk and ANOVA tests. The statistical software used to run these tests was IBM SPSS Statistics for Windows, version 21.0 (IBM Corp, Armong, New York).

RESULTS

The statistical analysis verified that LCU showed significance (p=0.004) with L1 (1.3±0.9) presenting Ra statistically greater than L3 (0.7±0.6), whereas L2 (1.0±0.8) showed intermediate values as seen in Table 3.

Time (p=0.705) and Solution (p=0.0884) factors and LCU x Solution (p=0.190), Time x LCU (p=0.911) and Time x Solution (p=0.089) interactions were not statistically significant. Surface roughness deltas were compared, observing that Solution showed significance (p=0.0042) with S2 (0.9±0.1) presenting statistically greater roughness than S3 (-0.1±0.1), whereas S1 (0.1±0.1) showed intermediate values, as seen in Table 3.

Table 3 | Mean values for surface roughness delta (Ra) by Light Curing (L) and Solution (S)

L1	L2	L3	S1	S2	S3
1.3*(±0.9)	1.0(±0.8)	0.7*(±0.6)	0.1(±0.1)	0.9*(±0.1)	-0.1*(±0.1)

*The mean difference is significant at the 0.05 level.

Time (p=0.691) and Light Curing (p=0.604) factors and Light Curing x Solution (p=0.080), Time x Light Curing (p=0.871), Time x Solution (p=0.184) and Time x Light Curing x Solution (0.438) interactions were not statistically significant.

Regarding color, statistical significance was observed for ΔE_{00} only for the LCU, where L1 (ΔE_{00} = 23.3±0.3) obtained lower ΔE_{00} than the other LCUs (ΔE_{00} = 24.7±24.7 for L2 and ΔE_{00} = 25.1±25.1 for L3) as seen in Table 4. No significant differences were found for Solution (p = 0.530), Time (p = 0.642) and Light Curing x Solution (p = 0.183), Light Curing x Time (p = 0.602), Solution x Time (p = 0.277) and Light Curing x Solution x Time (p = 0.4).

Table 4 | Color variation mean values (ΔE_{00}) by Light Curing

L1	L2	L3
23.3(±0.3)	24.7(±0.3)	25.1(±0.3)

Significant difference was also found for the C and H parameters. For C, the light curing L3 (ΔC' = 28.6±0.2) presented higher chroma compared to L1 (ΔC' = 27.8±0.2) and L2 (ΔC' = 27.5±0.2) (Table 5); solution S3 (ΔC' = 28.5±0.2) also presented higher chroma compared to the other solutions (ΔC' = 27.8±0.2 for S1 and ΔC' = 27.5±0.2 for S2), as seen in Table 5; and at T1 (ΔC' = 27.8±0.1) there was a lower chroma value, and this value increased over time (ΔC' = 28.0±0.1 for T2 and ΔC' = 28.1±0.1 for T3) (Table 5). When analyzing parameter H, it was found that light curing L1 (ΔH' = 53.1±0.1) promoted lower hue value compared to other curing protocols (ΔH' = 55.6±0.1 for L2 and ΔH' = 55.9±0.1 for L3), as seen in Table 6; and solution S3 (ΔH' = 55.4±0.1) also presented higher H value compared to S1 (ΔH' = 54.7±0.1) and S2 (ΔH' = 54.5±0.1) (Table 6).

Table 5 | Color variation mean values (ΔC') by Light Curing (L), Solution (S) and Time (T)

L1	L2	L3	S1	S2	S3	T1	T2	T3
27.8(±0.2)	27.5(±0.2)	28.6(±0.2)	27.8(±0.2)	27.6(±0.2)	28.5(±0.2)	27.8(±0.1)	28.0(±0.1)	28.1(±0.1)

Table 6 | Color variation mean values (ΔH') by Light Curing (L) and Solution (S)

L1	L2	L3	S1	S2	S3
53.1(±0.1)	55.6(±0.1)	55.9(±0.1)	54.7(±0.1)	54.5(±0.1)	55.4(±0.1)

DISCUSSION

Performance of GR strongly depends on the correct application of the LCU. Inadequate photopolymerization leads to marginal microinfiltration and to a degradation of the physical and mechanical properties of these materials.[7,9,10,19] Since antiseptic mouthwash is an element often used by patients, it is important to know its influence on the durability and resistance of the restoration materials used. Experimental studies should be performed to evaluate relevant properties of these materials, e.g., roughness and color. Roughness is involved in irregularities, prominences and crevices in the restoration material, all having great clinical impact in the retention of biofilm, color alterations and patient comfort. This study considers that these conditions can influence roughness and color and, consequently, in the clinical outcome of restorations.[11]

After performing the analysis of Ra, it was observed that LCU was a significant factor, with L1 (1.3±0.9) presenting statistically greater Ra than L3 (0.7±0.6) and L2 (1.0±0.8) having intermediate values. On the contrary, statistical significance was not observed for Time and Solution factors and LCU x Solution, Time x Light Curing and Time x Solution interactions.

All LCU tested in this study use LED-based technology. They differ in power output, requiring different exposure time for the restoration material.[20] Faster reactions of composite resins in the pre-gel phase can result in a reduced internal flow, consequently producing less deformation for non-attached surfaces and increasing the contraction due to polymerization. This can have a negative impact on the adhesive interface of the material. Studies show that exposure time to the LED curing interfere considerably on the tension of the restoration material.[20] Greater power output is desirable to obtain a greater conversion ratio of the monomers for greater mechanical properties in shorter times. Conversely, longer exposition of lower power outputs maintains marginal integrity with less polymerization contraction and surface roughness.[20]

Delta surface roughness analysis found significance for the Solution (p=0.042) factor, with S2 (p=0.184) presenting statistically greater roughness than S3 (p=-0.122) and S1 (p=0.086) having intermediate values. Time (p=0.691), Light Curing (p=0.871), Time x Solution (p=0.184) and Time x Light Curing x Solution (0.439) were not statistically significant.

Existing literature shows conflicting results regarding the influence of antiseptic mouthwash with and without alcohol in the analysis of the surface roughness of restoration materials. Some studies[21,22] show a negative influence of alcohol-based mouthwashes on surface roughness, related to a degradation of the restoration material caused by the pH in the solution. Contrary to this, Bohner et al.[23] found no influence by the same solution on the properties of the same restoration material. The effect on surface roughness will depend on the individual components of the material and the mouthwash solution. This is because the specimens immersed in the solutions received the same treatment in their preparation before being subjected to the chemical challenge regarding the care of the thickness of the resin increments and following the photopolymerization guidelines indicated by the manufacturers. Polymers are known to be very reactive and to react with substances to which they are continuously exposed.[24] These results reinforce the need for the continuous development of dental materials that are analyzed for their chemical stability to promote greater durability and survival of restorations.

Compared to CIE L* a* b* system advocated in previous years studies, the CIEDE2000 system uses the definitions and concepts of hue and chroma in a specific formula, confirming the importance of ideas and definitions suggested by Munsell. The values of ΔE_{00}, $\Delta L'$, $\Delta C'$ and $\Delta H'$ represent the differences of E', L', C' and H' between the current and initial readings at the beginning of the experiment.[25]

Regarding ΔE_{00}, significance was observed in test specimens that were photopolymerized by all light curing protocols, with Valo (ΔE_{00}=23.3±0.3) presenting lower color variation than the other two: Bluephase (ΔE_{00}=24.7±0.3) and Optilight Max (ΔE_{00}=25.1±0.3).

Given that L1 has higher power output than L2 and L3 and that the conversion ratio of composite resins is directly related to power output, the observed behavior can be justified by the fact that photoinitiators that are not photopolymerized can undergo vitrification and thus return to their original state, giving the material a yellow tint, contributing to color variation over time.[25,26] Instead, the yellow contributed by camphorquinone is not considered critical in the material used for this study due to its use in areas that are darker than those in which the composite resins are used and that tend to be more sensitive to color variations.[27]

Regarding the luminosity L', no statistical differences were found before and after the proposed treatments. Therefore, the composite resin remained stable after polymerization under different light curing protocols and immersion in different solutions.

It was observed that, after statistical analysis, the L3 curing induced higher Chroma (C') values compared to L1 and L2. Studies have reported that lower power light curing protocols promote a lower degree of resin conversion, inducing greater dye absorption and causing a significant change in Chroma. Because of this, the use of higher power curing light contributes to better restoration aesthetics.[28] Application time also influenced the numbers of $\Delta C'$. The respective values increased according to longer application time, negatively altering resin saturation.

Alcohol is used in mouthwash as a solvent, as a flavor intensifier and as an antiseptic agent. Great concern has been expressed on the use of alcohol as it can increase surface degradation in restoration materials.[16] Moreover, ethanol can reduce bonding

strength between the resin matrix and inorganic fillers, decreasing resilience to erosion and tainting the resin matrix.[11] Dash and Kallepalli[11] concluded that a material immersed in alcohol-based mouthwash resulted in decreased hardness when compared to the same material immersed in alcohol-free mouthwash. The results found so far were contradictory with the literature since mouthwashes did not promote color change. However, it was found that solution S3 promoted changes in ΔC' and H'. According to Arregui et al.,[29] water sorption is one of the factors associated with the discoloration of compounds. In addition, the structure of adhesive monomers influences the hydrophilicity of resulting polymers. Another factor associated with water sorption is the type and quantity (volume % weight %) of the filler particles used.

Hue can be defined as the natural color of tooth, derived from the internal dentin structure and determined by wavelength of light reflected by teeth. This study found that L1 promoted lower values of H'. Studies confirm that the higher the degree of resin conversion, the smaller the color change of material and, consequently, the lower the hue.[30]

According to Rüttermann et al.,[31] color stability depends on the quality of hardening, on the conversion of the photoinitiator system and on material composition. Moreover, during photopolymerization, initiators or synergists form subproducts that discolor when exposed to heat or ultraviolet light and alter the color of the resin increasing the red and yellow components.

Özdas et al.[32] state that possible color variations analyzed and observed in composite resins may be due to the type of composite (resin matrix, size and type of filler particles) associated to the type of solution and immersion time.

Existing literature shows instances that exhibit minimal color variation in control group specimens immersed in artificial saliva.[33] This is in line with the analysis made by Afzali et al.,[34] who observed no significant color variation in resins submitted to water, supporting the notion that water does not lead to chromatic alterations. In this study, specimens in the control group exposed to potable water showed no chromatic alteration.

Other studies analyzed the potential for tainting by liquids such as energy drinks[35] and mouthwash products.[36] Analysis of energy drinks showed that immersions of resins (Clearfil Majesty posterior, Filtek Supreme, Clearfil APX and Z250) during a six-month period presented color variation. According to this study, all solutions resulted in chromatic variation beyond what is considered clinically acceptable. Lepri et al.[36] evaluated some commonly used antiseptic mouthwashes (Plax, Listerine and Periogard) and found no significant color variation, inferring that their vibrant colorations do not have influence on the restoration materials used. Instead, an influence was observed in the luminosity of the resin with a minor negative impact on aesthetics.

Different light sources can significantly change the surface of photopolymerizable resins, including surface roughness, as shown by Trauth et al.[21] and Cengiz et al.,[22] in which they evaluated these changes in specimens when immersed in solutions. The lack of adequate polymerization makes the surface susceptible to alterations and the action of substances with different temperatures, pH and color. In this work, the specimens were activated with light sources at the time indicated by the manufacturer, so it can be attributed to the immersion and not by the absence of polymerization. Therefore, after an exploration of the existing literature and the *in vitro* nature of the proposed study, future studies, clinical or not, will help to further validate the methods used in the present study.

CONCLUSION

Light curing with the highest power output (L1) and the alcohol-free mouthwash (S2) lead to greater surface roughness. L1 also presented the lowest color variation.

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